Analysis of Amphetamine & Methamphetamine

1. Background

Lysergic acid diethylamide (LSD), commonly referred to as "acid", is a synthetic hallucinogen. LSD is manufactured from lysergic acid, which is found in ergot, a fungus that grows on rye and other grains. It is a colorless, odorless, and tasteless liquid. It comes in a variety of forms, but is always taken orally. LSD is most commonly found of small squares of paper called blotter (full sheet of paper are decorated with artwork or designs, perforated, then soaked in liquid solution and dried). Other forms include, tablets (microdots), gelatin squares (window panes), liquid, liquid sugar cubes and powder. Additionally, LSD has been embedded in candy such as "Gummy Worms," "Sweet Tarts," "Smarties," and "Pez."

2. Objective

The objective of this SOP is to establish guidelines to be used for the analysis of a sample that may contain amphetamine, methamphetamine and related phenethylamine.

3. Scope

This SOP is to be used by the laboratory staff of the Division of Analytical Chemistry at William A. Hinton State Laboratory Institute in Boston, MA.

{ DATE \@ "M/d/yyyy" }

4. Responsibility

Chemists are responsible for acquiring glassware, preparing chemical reagents and standards, sample analysis, and reporting. Chemists also perform instrument calibrations, maintenance and troubleshooting, ordering of supplies and other necessary tasks related to this analysis.

Laboratory Supervisors ensure that chemists are following this SOP. They may perform the duties of the chemists and must review raw data and reports generated by chemists. The Supervisor may advise the chemists of alternative testing methods. They ensure that quality control measures are within acceptable limits and determine when corrective actions are needed. They coordinate proficiency testing (PT), reporting and distribution of PT results. They oversee sample results distribution to outside agencies.

Directors ensure that the SOP is being followed and reviewed on a regular basis. They provide approval of standard operating procedures and review quality control documentations.

1. Related Documents

Cole, Michael, "The Analysis of Controlled Substances," London: John Wiley & Sons Ltd., 2003
Drug Enforcement Administration, "Basic Training Program for Forensic Drug Chemists," Drug Enforcement Administration.
Mills III, Terry et al, "Instrumental Data for Drug Analysis," 3rd ed., 6 vols., New York: CRC Press, 2006.
Moffat, A.C. et al, "Clarke's Isolation and Identification of Drugs," 2nd ed., London: The Pharmaceutical Press, 1986.
Moffat, A.C. et al. "Clarke's Analysis of Drugs and Poisons," 3rd ed., London: The Pharmaceutical Press, 2004.
Saferstein, Richard, "Forensic Science Handbook," New Jersey: Prentice Hall, 1988.
Scientific Working Group for the Analysis of Seized Drug Recommendation, 6th ed., "Part III A & B, Methods of Analysis/Sampling of Seized Drug for Qualitative Analysis," July 2011

6. Definitions

GC: Gas Chromatography
GC/MS: Gas Chromatography/Mass Spectrometry

7. Supplies, Equipment & Reagents

Supplies

Culture tubes
Spatula
Pasteur pipette
Volumetric Flask
Weighing dish
Weighing paper
GC vials with Teflon caps

Equipment

Analytical Balance
Ultraviolet (UV) Lamp
GC with FID

GC/MS

Reagents

p-dimethylaminobenzaldehyde (p-DMAB)
Lysergic acid diethylamide (LSD)
Lysergic acid methylpropylamide (LAMPA)
95% Ethanol
Concentrated Hydrochloric Acid
Methanol
Cocaine Hydrochloride
Codeine Phosphate
Chloroform
Sodium Bicarbonate
Tartaric Acid
Water

8. Safety

Due to the potential hazards, appropriate precautions should be taken as necessary. This includes, but is not limited to, the use of fume hoods, gloves, masks and safety glasses. Lab coats are to be worn at all times in the unit, unless performing administrative duties.

9. Reagent Preparation

Cobalt Thiocyanate Reagent
Dissolve 2.0g of cobalt thiocyanate in 100mL of deionized water. Mix the solution until completely dissolved.

Marquis Reagent
Dilute 10mL of 37% formaldehyde solution in 90mL of concentrated sulfuric acid. While stirring, slowly add the concentrated sulfuric acid to the formaldehyde solution. Allow the solution to cool completely.

Froedhde's Reagent
Dissolve 0.5g of sodium molybdate in 100mL of concentrated sulfuric acid. Mix the solution until completely dissolved.

Mecke's Reagent
Dissolve 1.0g of selenous acid in 100mL of concentrated sulfuric acid. Mix the solution until completely dissolved.

2.8N Hydrochloric Acid Reagent
Dilute 92.6mL of 12.1N hydrochloric acid in 400mL of deionized water. Mix the solution completely.

Cocaine/Codeine Standard (QC Mix)
Dissolve 10.0 mg of cocaine hydrochloride and 10.0mg of codeine phosphate and bring to volume with 10mL of methanol. Mix the solution until completely dissolved.

0.1N Sodium Hydroxide Reagent
Dissolved 0.8g of sodium hydroxide in 200mL of deionized water. Mix the solution until completely dissolved.

Amphetamine Standard

Methamphetamine Standard

10. Procedure

A. Document observations on the Drug Analysis Form noting the number, type (e.g. powder, crystalline or liquid) and marking of all items.
B. Sampling Plan
   i.
C. Color Test
   i. The color test consists of four reagents, which are cobalt thiocyanate, marquis, froehde's, and mecke's.
   ii. Place a couple of drops of cobalt thiocyanate, marquis, froehde's, mecke's reagents into individual wells on a porcelain spot plate.
   iii. Add a small amount of sample (1-2mg of powder or 1-2 drops of liquid) to each well.
   iv. Note any color change or reaction. The results will be recorded on the Drug Analysis Form by documenting the actual color/s observed. Negative observations will be recorded by stating no reaction present or no color change.
D. Interpretation
   i. Marquis reagent: Formation of an orange to brown color indicates the possible presence of amphetamine, methamphetamine, fentanyl or phentermine.
E. Ultraviolet/Visible Spectrophotometer
   i. Place about 1-2mg of sample into a labeled culture tube.
   ii. Add about 2mL of 0.1N sulfuric acid to the labeled culture tube and mix gently until dissolved.
   iii. Place 2-3mL of 0.1N sulfuric acid in a clean cuvette and place into the cuvette holder. Initiate the instrument to perform a blank analysis. (Blanks will be run between each unknown sample and reference standard.)
   iv. Place 2-3mL of ephedrine stock solution into a cuvette and place into the cuvette holder. Initiate the instrument to perform a standard analysis.
   v. Place 2-3mL of the unknown sample into a cuvette and place into the cuvette holder. Initiate the instrument to perform a sample analysis.
   vi. Print the spectra of all the unknown samples, reference standards and blanks.
   vii. Positive UV analysis will be recorded on the Drug Analysis Form by the use of a plus (+). The result is considered positive when the retention time of the sample and the reference standard (Cocaine/Codeine Mix) meet the laboratory criteria and are specified in the notes. Negative observations will be recorded by the use of a negative (-).
F. Base Conversion
   i. Place about 1-3mg of the sample into a labeled culture tube.
   ii. Add about 1-2mL of 0.1N sodium hydroxide into the labeled culture tube. _Allow the sample to solubilize for about 2-3 hours.
   iii. Add about 1-2mL of hexane to the labeled culture tube and mix gently.

iv. Remove the hexane layer from the solution. Transfer the extract into a labeled GC vial and cap tightly.

G. Gas Chromatography (as necessary)
   i. The extract from section (F) can be used for the GC analysis.
   ii. Initiate auto sampler sequence using the GENSCAN method running a blank solvent between each unknown sample and reference standard/s.
   iii. Compare retention time of the each sample with the reference standard/s. Also check the chromatograph to determine if the sample needs to be diluted or concentrated.
   iv. Positive GC analysis will be recorded on the Drug Analysis Form by the use of a plus (+). The result is considered positive when the retention time of the sample and the reference standard (Cocaine/Codeine Mix) meet the laboratory criteria and are specified in the notes. Negative observations will be recorded by the use of a negative (-).

H. Gas Chromatography/Mass Spectrometry
   i. Confirmatory analysis can be performed using the GC vial from the previous section (E).
   ii. Initiate auto sampler sequence using the SPEED method running a blank solvent between each unknown sample and reference standard/s.
   iii. Compare retention time and ion spectra of the each sample with the reference standard/s (Amphetamine or Methamphetamine).
   iv. Document the date analyzed and results of the GC/MS onto the MS Tracking Sheet, Drug Analysis Form and Control Card.

11. Documentation

A. All results will be documented on the Drug Analysis Form.
B. All raw data will be generated and filed according to the laboratory policy.
C. A certificate of analysis will be generated for each lab number which will document the results.

12. Attachments

GC Method

GC/MS Method